TEMPLATE EFFECT ON THE SYNTHESIS OF NANO WO_3 THIN FILM

QONITATILLAH AND MADZLAN AZIZ
Chemistry Department, Faculty of Science, UTM, 81310 Skudai, Johor, Malaysia

Introduction

Tungsten oxide(WO_3), which is one of the d^0-transition metal oxides, has many interesting physical and chemical properties which makes them useful for a number of thin film applications. The electrochromic properties of WO_3 and its hydrates were extensively investigated, which may be useful for applications in smart windows of displays [1], gas sensors, resist materials in ion beam lithography[2] and catalysis. The WO_3 films have been prepared by thermal evaporation[3],chemical vapour deposition[4], sputtering[5], spray pyrolisis [6] and sol-gel[7] method, the latter methodology being very attractive due to its low cost and to the formation of homogenous film. In this paper, WO_3 thin films were prepared by reacting tungstic acid with hydrogen peroxide and the presence of PEG as a template was studied.

Experimental

Materials

In this paper, the sol-gel route was presented for the preparation of the WO_3 sol. The solution was prepared by reacting tungstic acid powder(tungstic(VI)acid, Alfa Aesar) with hydrogen peroxide (30-32%, QRëC, grade AR) with 5% wt of WO_3. Poly ethylene glycol (PEG) 300 was obtained from Fluka and added into the solution.

Apparatus and Procedures

The solution was stirred for 24 hours using rod stirrer (IKA RW 20 digital) at 1000 rpm.After stirring, the solution was aged for another day and PEG (poly ethylene glycol) 300 was added into the solution for PEG:WO_3 = 0.25 and mixed for 8 hours aged overnight.The WO_3 films were deposited by the dip-coating method on glass plates (microscope slides,12Ω/slide) previously cleaned and rinsed with distilled water, acetone and then dried at 70ºC. The films were heated up to 70ºC in air and cooled before depositing other dips. The films then annealed in air to 300ºC for 1 hour at heat rate of 5ºC/minute.The surface morphological characteristics were examined using JEOL JSM 6701 F Field Emission Scanning Electron Microcope (FESEM). Vibrational spectroscopic studies for the films (as deposited and annealed at 300ºC) were carried out with Perkin Elmer Spectra One spectrophotometer (Veberlingen, Germany) in the frequency range of 4000-400 cm^{-1}. X-ray diffraction (XRD) pattern were recorded in the 2θ range from 20.0250º to 49.9750º for BB optic phase analysis. Cu K_α radiation was used ($K_{\alpha1}$ = 1.54060 Å, $K_{\alpha2}$ = 1.54443 Å, K_β = 1.39225 Å). Absorbancy (A) for both WO_3 solution and WO_3-PEG solution were recorded in the 260-500 nm wavelength range , in a UV-Vis Perkin Elmer lambda 25 version 2.85.04 spectrophotometer.

Result and discussion

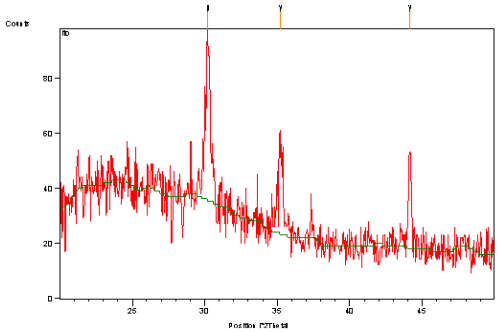

Fig.1 XRD profile of WO_3 film

Fig.1 shows the X-ray diffraction pattern of WO_3 film without PEG.It shows three strong peaks. The highest is at 2θ = 30.231 with height 56.06, the second is at 2θ = 44.1606 with height 35.75 and the last is at 2θ = 35.2259 with height 33.62. From the graphic, it seems that the particle of WO_3 is semicrystalline with three peaks on it.

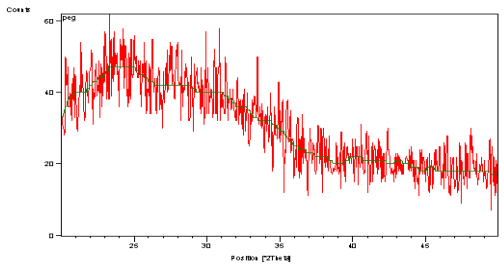

Fig.2 XRD profile of WO_3-PEG film

When PEG was added to the coating solution, the XRD data shows (from Fig.2) that the crystallization of WO_3 did not occurred.There is no sharp peak indicated a crystalline phase of WO_3 particle. It is correlated with FTIR data for WO_3-PEG indicated amorphous material from W-O-W stretching bands at 666.24 cm^{-1}.

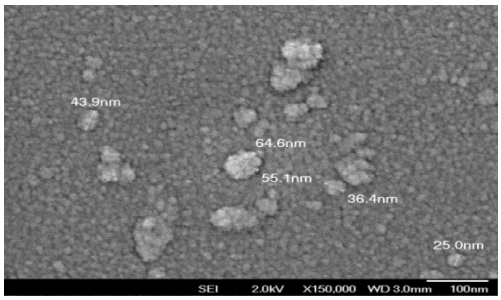

Fig.3 The surface morphology of WO_3

Fig. 3 shows field emission scanning electron micrograph of WO_3 film deposited using a solution of tungstic acid without PEG, annealed for 1 h at 300ºC. FESEM image has shown the morphology of the film is homogenous. The regular distribution of grains all over the surface is attributed to the uniform temperature maintained on the substrate. The particle size is quite regular, between 25.0 nm-64.6 nm, indicated as a nano size particle.

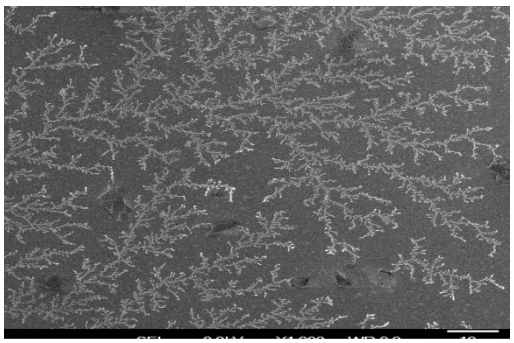

Fig.4The pattern of template on surface

In Fig. 4, it is shown that the surface has a unique pattern and WO_3 particle are dispersed along pattern. The presence of PEG directed the coating uniformly along the PEG's particles with particle size between 30.2-147.3 nm. The bigger size is due to the agglomeration of WO_3 and PEG.

Conclusion

We have synthesized nano particle WO_3 thin film using dip-coating method. The solution of tungstic acid stabilized by an organic additive acts as a template, PEG 300. The presence of template influenced the distribution, size and crystallinity of WO_3 particles. The template directed the spreading of WO_3 particles among substrate and followed the pattern of template and spreaded a long the PEG's particle.The size of particles are bigger because of agglomeration between WO_3 and PEG.The last, addition of PEG decreased the intensity of peak and indicated the formation of amorphous material.

References

[1]C.G. Granquist, *Handbook of Inorganic Eletrochromic Materials*, Elsevier, Amsterdam(1995)

[2] M.Penza and L.Vasanelli, SAW NO_x gas sensor using WO_3 thin film sensitive coating *Sensors and Actuators B.***41** (1997), p.31

[3] M.Penza, G.Cassano and F.Tortorella,Gas recognition by activated WO_3 thin film sensors array *Sens.Actuators B:Chem.***81**(2001), p.115-121

[4] A.D.Kuypers, C.I.M.A.Spee, J.L.Linden, G.Kirchner, J.F.Forsyth and A.Mackor,Plasma enhanced CVD of electrochromic materials, *Surf.Coat.Tech.***7/75** (1995), p.1033-1037

[5] A.Monteiro, M.F.Costa, B.Almeida, V.Tixeira, J.Gago and E.Roman,Structural and optical characterization of WO_3 deposited on glass and ITO, *Vacuum* **64** (2002), p 287-291

[6] M.Regragui, V.Jousseaume, M.Addou,A.Outzourhit, J.C.Bernede and E.El Idrissi, Electrical and optical properties of WO_3 thin films.*Thin Solid Films* **397** (2001), p.238-243

[7] M.Tong, G.Dai and D.Gao,WO_3 thin film sensor prepared by sol-gel technique and its low temperature sensing properties to trimethylamine, *Mater.Chem.Phys.***69** (2001), p.176-179